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## **DRES Database of Methods for the Analysis of Chemical Warfare Agents**

BY

Paul. A. D'Agostino, Lionel R. Provost & James R. Hancock

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ABSTRACT

DRES analytical methods have been published regularly in the open literature along with the methods developed by others for CW agent sample preparation and analysis. Several years ago the key bibliographic information from this database of papers was entered into Procite, a searchable bibliographic database program. Update of the database continues as an ongoing effort and the DRES Database of Methods for the Analysis of Chemical Warfare Agents is available in hardcopy form or as a softcopy Procite or Wordperfect file. One hundred and fifty literature entries have been made as of May 1997.

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### INTRODUCTION

The Canadian Forces may be called on to perform peacekeeping or battlefield operations in regions of the world where there is a significant threat of chemical warfare (CW) agent use. To operate effectively in these theatres the CF must be able to identify the chemical warfare agent(s) being used. Development of instrumental analytical methods for the identification and confirmation of these compounds is an important CF requirement that is addressed by DRES analytical researchers. Mass spectrometry (MS) and gas and liquid chromatography (GC and LC), core capability areas in the analytical sciences at DRES, form the basis for current analytical methods for the identification and confirmation of CW agents. In-house methods have been validated during NATO and UN analytical exercises with improvement continuing through the exploitation of new analytical technologies such as electrospray mass spectrometry and capillary electrophoresis.

DRES analytical methods have been published regularly in the open literature along with the methods developed by others for CW agent sample preparation and analysis. Several years ago the key bibliographic information from this database of papers was entered into Procite, a searchable bibliographic database program. Update of the database continues as an ongoing effort and the DRES Database of Methods for the Analysis of Chemical Warfare Agents is available to TTCP panel members in hardcopy form or as a softcopy Procite or Wordperfect file. One hundred and fifty literature entries have been made as of May 1997, with the complete list following alphabetically.

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**DRES Database of Methods for the Analysis of Chemical Warfare Agents**

1. Albro, P. W. and L. Fishbein. "Gas Chromatography of Sulfur Mustard and its Analogues" in *J. Chromatogr.* 1970, 46:202-203.
2. Alfthan, K., H. Kenttamaa and T. Zukale. "Characterization and Semiquantitative Estimation of Organophosphorus Compounds Based on Inhibition of Cholinesterases" in *Analytica Chimica Acta*. 1989, 217:43-51.
3. Ali-Mattila, E., K. Siivinen, H. Kenttamaa and P. Savolahti. "Mass Spectrometric Methods in Structural Analysis of Some Vesicants" in *Int. J. Mass Spectrom. Ion Phys.* 1983, 47:371-374.
4. Andersson, G. "Analysis of Two Chemical Weapons Samples from the Iran/Iraq War" in *NBC Defence and Technology International*. 1986, April:62-65.
5. Appler, B. and K. Christmann. "Detection of B,B'-Dichloroethyl Sulfide on Thin-Layer Chromatograms" in *J. Chromatogr.* 1983, 264:445-452.
6. Avdovich, H. W., A. By, J. C. Ethier and G. A. Neville. "Spectral Identification of a Lachrymatory Exhibit as CS" in *J. Can. Soc. Forens. Sci.* 1981, 14:172-178.
7. Benschop, H. P., E. C. Bijleveld, M. F. Otto, C. E. A. M. Degenhardt, H. P. M. Van Helden and L. P. A. De Jong. "Stabilization and Gas Chromatographic Analysis of the Four Stereoisomers of 1,2,2-Trimethylpropyl Methylphosphonofluoridate (Soman) in Rat Blood" in *Anal. Biochem.* 1985, 151:242-253.

8. Benschop, H. P. and L. P. A. De Jong. "Nerve Agent Stereoisomers: Analysis, Isolation, and Toxicology" in *Acc. Chem. Res.* 1988, 21:368-374.
9. Bhattacharya, A. and D. N. Tripathi. "Field Desorption Mass Spectra of Pyridinium Oxime Salts with Rapid Heated Emitter" in *Anal. Chem.* 1984, 56:2295-2297.
10. Black, R. M. "Tandem Mass Spectrometry: Applications in the Trace Analysis of Chemical Warfare Agents" in *J. Defence Sci.* 1996, 1:219-226.
11. Black, R. M., K. Brewster, R. J. Clarke and J. M. Harrison. "The Chemistry of 1,1'-Thiobis(2-chloroethane) (Sulphur Mustard) Part II. The Synthesis of Some Conjugates with Cysteine, n-Acetylcysteine and n-Acetylcysteine Methyl Ester" in *Phosphorus, Sulfur, and Silicone.* 1992, 71:49-58.
12. Black, R. M., K. Brewster, J. M. Harrison and N. Stansfield. "The Chemistry of 1,1'-Thiobis(2-chloroethane) (Sulphur Mustard) Part 1. Some Simple Derivatives" in *Phosphorus, Sulfur, and Silicon.* 1992, 71:31-47.
13. Black, R. M., R. J. Clarke and R. W. Read. "Analysis of 1,1'-Sulphonylbis[2-(methylsulphinyl)ethane] and 1-Methylsulphinyl-2-[2-(methylthio)ethylsulphonyl]ethane, Metabolites of Sulphur Mustard, in Urine using Gas Chromatography-Mass Spectrometry" in *J. Chromatogr.* 1991, 558:405-414.
14. Black, R. M., R. J. Clarke, D. B. Cooper, R. W. Read and D. Utley. "Application of Head Space Analysis, Solvent Extraction, Thermal Desorption and Gas Chromatography-Mass Spectrometry to the Analysis of Chemical Warfare Samples Containing Sulphur Mustard and Related Compounds" in *J. Chromatogr.* 1993, 637:71-80.

15. Black, R. M., R. J. Clarke, R. W. Read and M. T. J. Reid. "Application of Gas Chromatography-Mass Spectrometry and Gas Chromatography-Tandem Mass Spectrometry to the Analysis of Chemical Warfare Samples Found to Contain Residues of the Nerve Agent Sarin, Sulphur Mustard and their Degradation Products" in *J. Chromatogr. A*. 1994, 662:301-321.
16. Black, R. M. and R. W. Read. "Detection of Trace Levels of Thiodiglycol in Blood, Plasma and Urine Using Gas Chromatography-Electron-Capture Negative-Ion Chemical Ionisation Mass Spectrometry" in *J. Chromatogr.* 1988, 449:261-270.
17. Black, R. M. and R. W. Read. "Methods for the Analysis of Thiodiglycol Sulfoxide, a Metabolite of Sulfur Mustard, in Urine using Gas Chromatography-Mass Spectrometry" in *J. Chromatogr.* 1991, 558:393-404.
18. Borrett, V. T., R. Colton and J. C. Traeger. "The Electrospray Mass Spectra of Phosphonic Acid, Methyl Phosphonic Acid and its Alkyl Esters, and their Complexes with Alkali and Alkali Earth metal ions" in *Eur. Mass Spectrom.* 1995, 1:131-140.
19. Borrett, V. T., R. J. Mathews and E. R. Mattsson. "Verification of the Chemical Weapons Convention: Mass Spectrometry of Alkyl Methylphosphonofluoridates" in *Aust. J. Chem.* 1994, 47:2065-2074.
20. Borrett, V. T., R. J. Mathews, R. Colton and J. C. Traeger. "Verification of the United Nations Chemical Weapons Convention: the Application of Electrospray Mass Spectrometry" in *Rapid Commun. Mass Spectrom.* 1996, 10:114-118.

21. Bossle, P. C., D. J. Reutter and E. W. Sarver. "Analysis of Alkyl Methylphosphonic Acids in Aqueous Matrices by Ion-Pair Reverse-Phase Ion Chromatography" in *J. Chromatogr.* 1987, 407:399-404.
22. Braue Jr., E. H. and M. G. Pannella. "CIRCLE CELL FT-IR Analysis of Chemical Warfare Agents in Aqueous Solutions" in *Applied Spectroscopy*. 1990, 44:1513-1520.
23. Camel, V., M. Caude and A. Tambute. "SFE of an Organophosphorous Compound from Soils with Capillary GC Analysis" in *J. Chromatogr. Sci.* 1995, 33:123-132.
24. Casselman, A. A., N. C. C. Gibson and R. A. B. Bannard. "A Rapid, Sensitive, Gas-Liquid Chromatographic Method for the Analysis of Bis(2-chloroethyl)sulfide Collected from Air in Hydrocarbon Solvents" in *J. Chromatogr.* 1973, 78:317-322.
25. Chou, C-C and S. R. Long. "Chemical Ionization Fourier Transform Mass Spectrometry of Chemical Warfare Agent Simulants using Laser-Produced Metal Ions" in *Applied Optics*. 1990, 29:4981-4986.
26. Clark, A. J. "Determination of Organosulfur Compounds and Amino Acid-Mustard Conjugates by Liquid Chromatography with Amperometric Detection" in *Anal. Proceedings*. 1993, 30:355-357.
27. D'Agostino, P. A. "Chemical Warfare Agents" in *Encyclopedia of Analytical Science*, Academic Press, 1995, 599-608.
28. D'Agostino, P. A., A. S. Hansen, P. A. Lockwood and L. R. Provost. "Capillary Column Gas Chromatography-Mass Spectrometry of Tabun" in *J. Chromatogr.* 1985, 347:257-266.



29. D'Agostino, P. A. and C. J. Porter. "Capillary Column Gas Chromatography/Tandem Mass Spectrometry Verification of Chemical Warfare Agents" in *Rapid Commun. Mass Spectrom.* 1992, 6:717-718.
30. D'Agostino, P. A., L. R. Provost and J. Visentini. "Analysis of O-Ethyl S-[2-(diisopropylamino)-ethyl] Methylphosphonothiolate (VX) by Capillary Column Gas Chromatography-Mass Spectrometry" in *J. Chromatogr.* 1987, 402:221-232.
31. D'Agostino, P. A. and L. R. Provost. "Analysis of Irritants by Capillary Column Gas Chromatography Tandem Mass Spectrometry" in *J Chromatogr. A.* 1995, 695:65-73.
32. D'Agostino, P. A. and L. R. Provost. "Capillary Column Ammonia Chemical Ionization Mass Spectrometry of Organophosphorus Chemical Warfare Agents and Simulants" in *Biomed. Environ. Mass Spectrom.* 1986, 13:231-236.
33. D'Agostino, P. A. and L. R. Provost. "Capillary Column Isobutane Chemical Ionization Mass Spectrometry of Mustard and Related Compounds" in *Biomed. Environ. Mass Spectrom.* 1988, 15:553-564.
34. D'Agostino, P. A., L. R. Provost, J. F. Anacleto and P. W. Brooks. "Capillary Column Gas Chromatography-Mass Spectrometry and Gas Chromatography-Tandem Mass Spectrometry Detection of Chemical Warfare Agents in a Complex Airborne Matrix" in *J. Chromatogr.* 1990, 504:259-268.
35. D'Agostino, P. A. and L. R. Provost. "Capillary Column Gas Chromatography-Ammonia and Deuterated Ammonia Chemical Ionization Mass Spectrometry of Sulfur Vesicants" in *J. Chromatogr.* 1992, 600:267-272.

36. D'Agostino, P. A. and L. R. Provost. "Capillary Column Electron Impact and Ammonia Chemical Ionization Gas Chromatographic-Mass Spectrometric and Gas Chromatographic-Tandem Mass Spectrometric Analysis of Mustard Hydrolysis Products" in *J. Chromatogr.* 1993, 645:283-292.
37. D'Agostino, P. A. and L. R. Provost. "Capillary Column Gas Chromatographic-Tandem Mass Spectrometric Analysis of Phosphate Esters in the Presence of Interfering Hydrocarbons" in *J. Chromatogr. A.* 1994, 670:127-134.
38. D'Agostino, P. A. and L. R. Provost. "Detection of Sarin and Soman in a Complex Airborne Matrix by Capillary Column Ammonia Chemical Ionization-Mass Spectrometry and Gas Chromatography-Tandem Mass Spectrometry" in *J. Chromatogr.* 1991, 541:121-130.
39. D'Agostino, P. A. and L. R. Provost. "Determination of Chemical Warfare Agents, their Hydrolysis Products and Related Compounds in Soil" in *J. Chromatogr.* 1992, 589:287-294.
40. D'Agostino, P. A., L. R. Provost, J. R. Hancock and C. A. Boulet. "Electrospray Mass Spectrometric Characterization of Six Therapeutic Oximes: HI-6, HS-6, Obidoxime, 2-PAM, TMB-4 and HLo-7" in *Rapid Commun. Mass Spectrom.* 1996, 10:805-810.
41. D'Agostino, P. A. and L. R. Provost. "Gas Chromatographic Retention Indices of Chemical Warfare Agents and Simulants" in *J. Chromatogr.* 1985, 331:47-54.
42. D'Agostino, P. A. and L. R. Provost. "Gas Chromatographic Retention Indices of Sulfur Vesicants and Related Compounds" in *J. Chromatogr.* 1988, 436:399-411.

43. D'Agostino, P. A., L. R. Provost and K. M. Looye. "Identification of Tabun Impurities by Combined Capillary Column Gas Chromatography-Mass Spectrometry" in *J. Chromatogr.* 1989, 465:271-283.
44. D'Agostino, P. A., L. R. Provost, A. S. Hansen and G. A. Luoma. "Identification of Mustard Related Compounds in Aqueous Samples by Gas Chromatography/Mass Spectrometry" in *Biomed. Environ. Mass Spectrom.* 1989, 18:484-491.
45. D'Agostino, P. A. and L. R. Provost. "Mass Spectrometric Identification of Products Formed During Degradation of Ethyl Dimethylphosphoramidocyanide (Tabun)" in *J. Chromatogr.* 1992, 598:89-95.
46. Dang, T. A., R. J. Day and D. M. Hercules. "Laser Mass Spectrometry of Diquaternary Ammonium Salts" in *Anal. Chem.* 1984, 56:866-871.
47. De Bisschop, H. C. and E. Michiels. "Assay of the Nerve Agent Soman in Serum by Capillary Gas Chromatography with Nitrogen-Phosphorus Detection and Splitless Injection" in *Chromatographia.* 1984, 18:433-436.
48. De Jong, L. P. A., E. C. Bijleveld, C. Van Dijk and H. P. Benschop. "Assay of the Chiral Organophosphate, Soman, in Biological Samples" in *Intern. J. Environ. Anal. Chem.* 1987, 29:179-197.
49. Degenhardt-Langelaan, C. E. A. M. and Ch. E. Kientz. "Capillary Gas Chromatographic Analysis of Nerve Agents using Large Volumn Injections" in *J. Chromatogr. A.* 1996, 723:210-214.

50. Degenhardt, C. E. A. M., A. Verweij and H. P. Benschop. "Gas Chromatography of Organophosphorus Compounds on Chiral Stationary Phases" in *Intern. J. Environ. Anal. Chem.* 1987, 30:15-28.
51. Donovan, W. H. and G. R. Famini. "Using Theoretical Descriptions in Structure Activity Relationships: Retention Indices of Sulfur Vesicants and Related Compounds" in *J. Chem. Soc., Perkin Trans. 2.* 1995, 83-89.
52. Ember, L. "Chemical Weapons: Residues Verify Iraqi use on Kurds" in *Chem. Eng. News.* 1993, May:8-9.
53. Epstein, J., J. J. Callahan and V. E. Bauer. "The Kinetics and Mechanisms of Hydrolysis of Phosphonothiolates in Dilute Aqueous Solution" in *Phosphorus.* 1974, 4:157-163.
54. Erickson, R. L., R. N. Macnair, R. H. Brown and H. D. Hogan. "Determination of Bis(2-chloroethyl)sulfide in a Dawson Apparatus by Gas Chromatography" in *Anal. Chem.* 1972, 44:1040-1041.
55. Ferslew, K. E., R. H. Orcutt and A. N. Hagardorn. "Spectral Differentiation and Gas Chromatographic/Mass Spectrometric Analysis of the Lacrimators 2-Chloroacetophenone and o-Chlorobenzylidene Malononitrile" in *J. Forensic Science.* 1986, 31:658-665.
56. Fowler, W. K. and J. E. Smith Jr. "Indirect Determination of O-Ethyl S-(2-diisopropylaminoethyl) Methylphosphonothioate in Air at Low Concentrations" in *J. Chromatogr.* 1989, 478:51-61.

57. Fowler, W. K. and J. E. Smith Jr. "Solid Sorbent Collection and Gas Chromatographic Determination of Bis(2-chloroethyl)sulfide in Air at Trace Concentrations" in *J. Chromatogr. Sci.* 1990, 28:118-122.
58. Fowler, W. K., D. C. Steward and D. S. Weinberg. "Gas Chromatographic Determination of Lewisite Hydrolysate, 2-Chlorovinylarsonous Acid, after Derivatization with 1,2-Ethanedithiol" in *J. Chromatogr.* 1991, 558:235-246.
59. Fredriksson, S-A, L-G Hammarstrom, L. Henriksson and H-A Lakso. "Trace Determination of Alkyl Methylphosphonic Acids in Environmental and Biological Samples Using Gas Chromatography/Negative-ion Chemical Ionization Mass Spectrometry and Tandem Mass Spectrometry" in *J. Mass Spectrom.* 1995, 30:1133-1143.
60. Gandhe, B. R., R. C. Malhotra and P. K. Gutch. "Gas Chromatographic Retention Indices of Tear Gases on Capillary Columns" in *J. Chromatogr.* 1989, 479:165-169.
61. Gibson, N. C. C., A. A. Casselman and R. A. B. Bannard. "An Improved Gas-Liquid Chromatographic Method for the Analysis of Bis(2-chloroethyl)sulfide Collected from Air by Solvent Entrapment" in *J. Chromatogr.* 1974, 92:162-165.
62. Griest, W. H., R. S. Ramsey, C-H Ho and W. M. Caldwell. "Supercritical Fluid Extraction of Chemical Warfare Agent Simulants from Soil" in *J. Chromatogr.* 1992, 600:273-277.
63. Hakkinen, V. M. A. "Analysis of Chemical Warfare Agents in Water by Solid Phase Extraction and Two-Channel Capillary Gas Chromatography" in *J. High Resoln. Chromatogr.* 1991, 14:811-815.

64. Hancock, J. R., J. M. McAndless and R. P. Hicken. "A Solid Adsorbent Based System for the Sampling and Analysis of Organic Compounds in Air: An Application to Compounds of Chemical Defence Interest" in *J. Chromatogr. Sci.* 1991, 29:40-45.
65. Hancock, J. R. and G. R. Peters. "Retention Index Monitoring of Compounds of Chemical Defence Interest Using Thermal Desorption Gas Chromatography" in *J. Chromatogr.* 1991, 538:249-257.
66. Hassan, S. S. M., J. M. Abdalla and N. E. Nashed. "Characterization and Determination of Benzalmalonitriles using Infared, Nuclear Magnetic Resonance and Mass Spectrometry" in *Mikrochimica Acta.* 1984, II:27-38.
67. Hesso, A. and R. Kostiainen. "Tandem Mass Spectrometry: A Potential Method for Detection and Identification of Chemical Warfare Agents" in *Proc. 2nd Int. Symp. Protection Against Chemical Warfare Agents, Stockholm, Sweden, 15-19 June 1986.* 1986, 257-260.
68. Heyndrickx, A., J. Cordonnier and A. De Bock. "Chromatographic Procedures for the Toxicological Determination of Bis(2-chloroethyl) Sulfide (Mustard Gas, Yperite) in Enviromental and Human Biological Samples" in *Arch. Belg. Med. Soc. (Toxicol.).* 1984, 102-109.
69. Huber, J. F. K., E. Kenndler, G. Reich, W. Hack and J. Wolf. "Optimal Selection of Gas Chromatographic Columns for the Analytical Control of Chemical Warfare Agents by Application of Information Theory to Retention Data" in *Anal. Chem.* 1993, 65:2903-2906.
70. Hutchinson, R. and J. Razulis. "Methodology Assessment for the Chemical Weapons Convention" in *Army RD&A.* 1996, May-June:29-30.

71. Ingram, J. C., G. S. Groenewold, A. D. Appelhans, J. E. Delmore and D. A. Dahl. "Detection of Alkylmethylphosphonic Acids on Leaf Surfaces by Static Secondary Ion Mass Spectrometry" in *Anal. Chem.* 1995, 67:187-195.
72. Jakubowski, E. M., C. L. Woodard, M. M. Mershon and T. W. Dolzine. "Quantification of Thiodiglycol in Urine by Electron Ionization Gas Chromatography-Mass Spectrometry" in *J. Chromatogr.* 1990, 528:184-190.
73. Kaaijk, J. and C. Frijlink. "Degradation of S-2-Di-Isopropylaminoethyl O-Ethyl Methylphosphonothioate in Soil. Sulphur-containing Products" in *Pestic. Sci.* 1977, 8:510-514.
74. Kaipainen, A., O. Kostainen and M-L Riekkola. "Identification of Chemical Warfare Agents in Air Samples using Capillary Column Gas Chromatography with Three Simultaneous Detectors" in *J. Microcol. Sep.* 1992, 4:245-251.
75. Ketkar, S. N., S. M. Penn and W. L. Fite. "Real-Time Detection of Parts per Trillion Levels of Chemical Warfare Agents in Ambient Air Using Atmospheric Pressure Ionization Tandem Quadrupole Mass Spectrometry" in *Anal. Chem.* 1991, 63:457-459.
76. Kientz, C. E., A. Verweij, G. J. de Jong and U. A. Th Brinkman. "Verification of Nonproduction of Chemical Warfare Agents: I. Determination of Organophosphorus Compounds by Microcolumn Liquid Chromatography with Flame Photometric or Thermionic Detection" in *J. Microcol. Sep.* 1992, 4:465-475.
77. Kientz, C. E., A. Verweij, G. J. de Jong and U. A. Th Brinkman. "Verification of Nonproduction of Chemical Warfare Agents: II. Large Volume Injections in Microcolumn Liquid Chromatography Using Flame Photometric Detection" in *J. Microcol. Sep.* 1992, 4:477-483.

78. Kokko, M. "Effects of Variations in Gas Chromatographic Conditions on the Linear Retention Indices of Selected Chemical Warfare Agents" in *J. Chromatogr.* 1993, 630:231-249.
79. Kolla, P. "Detecting Hidden Explosives" in *Anal. Chem.* 1995, 67:184A-189A.
80. Kostianinen, R., A. P. Bruins and V. M. A. Hakkinen. "Identification of Degradation Products of Some Chemical Warfare Agents by Capillary Electrophoresis-Ion Spray Mass Spectrometry" in *J. Chromatogr.* 1993, 634:113-118.
81. Kuitunen, M-L, K. Hartonen and M-L Riekkola. "Analysis of Chemical Warfare Agents in Soil Samples by Off-Line Supercritical Fluid Extraction and Capillary Gas Chromatography" in *J. Microcol. Sep.* 1991, 3:505-512.
82. Kunkel, G. J., K. L. Busch, R. Dunphy, D. J. Burinsky, R. Barak, P. Bel, G. Amitai and A. Vincze. "Liquid Secondary Ion Mass Spectra and Fast Atom Bombardment Mass Spectra of Diquaternary Pyridinium Oxime Salts" in *J. Mass Spectrom.* 1995, 30:282-289.
83. Lakkisto, U-M. "Retention Spectrometry - A New Method for Rapid and Reliable Detection and Identification of Chemical Warfare Agents" in *Proc. 2nd Int. Symp. Protection Against Chemical Warfare Agents, Stockholm, Sweden, 15-19 June 1986.* 1986, 245-250.
84. Leadbeater, L., G. L. Sainsbury and D. Utley. "ortho-Chlorobenzylmalononitrile: A Metabolite Formed from ortho-Chlorobenzylidenemalononitrile (CS)" in *Toxicology and Applied Pharmacology.* 1973, 25:111-116.



85. Lightenstein, D. A., E. R. J. Wils, S. P. Kossen and A. G. Hulst. "Identification of Two Metabolites of the Cholinestase Reactivator HI-6 Isolated from Rat Urine" in *J. Pharm. Pharmacol.* 1987, 39:17-23.
86. Machata, G. and W. Vycudilik. "Detection of Mustard Gas in Biological Material" in *Arch. Belg. Med. Soc. (Toxicol.)*. 1984, 53-55.
87. Maisonneuve, A., I. Callebat, L. Debordes and L. Coppet. "Specific and Sensitive Quantitation of 2,2'-Dichlorodiethyl Sulphide (sulphur mustard) in Water, Plasma and Blood: Application to Toxicokinetic Study in the Rat after Intravenous Intoxication" in *J. Chromatogr.* 1992, 583:155-165.
88. Martz, R. M., D. J. Reutter and L. D. Lasswell III. "A Comparison of Ionization Techniques for Gas Chromatography/Mass Spectroscopy Analysis of Dye and Lachrymator Residues from Exploding Bank Security Devices" in *J. Forensic Sci.* 1983, 28:200-207.
89. Massil, S. E. and D. Ovadia. "Determination of Phosgene as its N,N,N',N'-tetraethylurea Derivative by Gas Chromatography" in *J. Chromatogr.* 1991, 538:435-440.
90. Mazurek, M. and Z. Witkiewicz. "The Analysis of Organophosphorus Warfare Agents in the Presence of Pesticides by Overpressure Thin Layer Chromatography" in *J. Planar Chromatogr.* 1991, 4:379-384.
91. McAndless, J. M. and J. R. Hancock. "An Automated Air Sampling and Analysis System Based on Miniature Solid-Sorbent Tubes (Minitubes)" in *Proc. 2nd Int. Symp. Protection Against Chemical Warfare Agents, Stockholm, Sweden, 15-19 June 1986*. 1986, 181-187.

92. Mercier, J-P, Ph. Morin, M. Dreux and A. Tambute. "Capillary Electrophoresis Analysis of Chemical Warfare Agent Breakdown Products I. Counterelectroosmotic Separation of Alkylphosphonic Acids and their Monoester Derivatives" in *J. Chromatogr. A*. 1996, 741:279-285.
93. Mesilaakso, M. and E-L Tolppa. "Detection of Trace Amounts of Chemical Warfare Agents and Related Compounds in Rubber, Paint, and Soil Samples by  $^1\text{H}$  and  $^{31}\text{P}$   $\{^1\text{H}\}$  NMR Spectroscopy" in *Anal. Chem.* 1996, 68:2313-2318.
94. Munavalli, S., E. M. Jakubowski and H. D. Durst. "Liquid Chromatography/Thermospray Mass Spectrometry of Mustard and its Metabolites" in *J. Mass Spectrom.* 1995, 30:1716-1722.
95. Munavalli, S. and M. Pannella. "Thin-Layer Chromatography of Mustard and its Metabolites" in *J. Chromatogr.* 1988, 437:423-428.
96. Nowicki, J. "Analysis of Chemical Protection Sprays by Gas Chromatography/Mass Spectrometry" in *J. Forensic Sciences*. 1982, 27:704-709.
97. Preston, J. M., F. W. Karasek and S. H. Kim. "Plasma Chromatography of Phosphorus Esters" in *Anal. Chem.* 1977, 49:1746-1750.
98. Purdon, J. G., J. G. Pagotto and R. K. Miller. "Preparation, Stability and Quantitative Analysis by Gas Chromatography and Gas Chromatography-Electron Impact Mass Spectrometry of tert-Butyldimethylsilyl Derivatives of some Alkylphosphonic and Alkyl Methylphosphonic Acids" in *J. Chromatogr.* 1989, 475:261-272.

99. Raghuveeran, C. D. and R. C. Malhotra. "Reversed-Phase High-Performance Liquid Chromatography of some Irritants" in *J. Chromatogr.* 1982, 240:243-246.
100. Raghuveeran, C. D., R. C. Malhotra and R. S. Dangi. "Reversed-Phase High-Performance Liquid Chromatography of Sulphur Mustard in Water" in *J. Liq. Chromatogr.* 1993, 16:1615-1624.
101. Rezai, M. A., G. Famiglini and A. Cappiello. "Enhanced Detection Sensitivity by Large Volume Injection in Reversed-Phase Micro-High-Performance Liquid Chromatography" in *J. Chromatogr. A.* 1996, 742:69-78.
102. Sass, S. and T. L. Fisher. "Chemical Ionization and Electron Impact Mass Spectrometry of some Organophosphonate Compounds" in *Org. Mass Spectrom.* 1979, 14:257-264.
103. Sass, S., T. L. Fisher, R. J. Steger and G. A. Parker. "Gas Chromatographic Methods for the Analysis of Trace Quantities of Isopropyl Methylphosphonofluoridate and Associated Compounds, in situ and in Decontamination Effluent" in *J. Chromatogr.* 1982, 238:445-456.
104. Sass, S. and W. D. Ludemann. "Thin-Layer Chromatography of Phosphonic Acids" in *J. Chromatogr.* 1980, 187:447-452.
105. Sass, S. and G. A. Parker. "Structure-Response Relationship of Gas Chromatography-Flame Photometric Detection of some Organophosphorus Compounds" in *J. Chromatogr.* 1980, 189:331-349.

106. Sass, S. and R. J. Steger. "Gas Chromatographic Differentiation and Estimation of some Sulfur and Nitrogen Mustards using a Multidetector Technique" in *J. Chromatogr.* 1982, 238:121-132.
107. Sass, S. and M. H. Stutz. "Thin-Layer Chromatography of some Sulfur and Nitrogen Mustards" in *J. Chromatogr.* 1981, 213:173-176.
108. Schoene, K., J. Steinhilber, H. J. Bruckert and A. König. "Speciation of Arsenic-containing Chemical Warfare Agents by Gas Chromatography Analysis after Derivatization with Thioglycolic Acid Methyl Ester" in *J. Chromatogr.* 1992, 605:257-262.
109. Shih, M. L. and R. I. Ellin. "Determination of Toxic Organophosphorus Compounds by Specific and Nonspecific Detectors" in *Anal. Letters.* 1986, 19:2197-2205.
110. Shih, M. L., J. R. Smith, J. D. McMonagle, T. W. Dolzine and V. C. Gresham. "Detection of Metabolites of Toxic Alkylmethylphosphonates in Biological Samples" in *Bio. Mass Spectrom.* 1991, 20:717-723.
111. Singh, A. K., R. J. Zeleznikar Jr. and L. R. Drewes. "Analysis of Soman and Sarin in Blood Utilizing a Sensitive Gas Chromatography-Mass Spectrometry Method" in *J. Chromatogr.* 1985, 324:163-172.
112. Sipponen, K. B. "Detector for Organophosphorus Compounds in Liquid Chromatography Based on the Cholinesterase Inhibition Reaction" in *J. Chromatogr.* 1987, 389:87-94.

113. Smith, J. R. and J. J. Schlager. "Gas Chromatographic Separation of the Stereoisomers of Organophosphorus Chemical Warfare Agents Using Cyclodextrin Capillary Columns" in *J.High Resol. Chromatogr.* 1996, 19:151-154.
114. Smith, S. J. "Detection Methods for Highly Toxic Organophosphonates" in *Talanta*. 1983, 30:725-739.
115. Soderstrom, M. T., H. Bjork, V. M. A. Hakkinen, O. Kostianen, M-L Kuitunen and M. Rautio. "Identification of Compounds Relevant to the Chemical Weapons Convention using Selective Gas Chromatography Detectors, Gas Chromatography-Mass Spectrometry and Gas Chromatography-Fourier Transform Infrared Spectroscopy in an International Trial Proficiency Test" in *J. Chromatogr. A*. 1996, 742:191-203.
116. Sokolowski, M. and J. K. Rozylo. "TLC Analysis of Warfare Agents under Battlefield Conditions" in *J. Planar Chromatogr.* 1993, 6:467-471.
117. Sokolowski, M. and Z. Witkiewicz. "Gas Chromatography-Mass Spectrometry Analysis of Products of o-Isopropyl Methylphosphonofluoridate Transformation in Aliphatic Alcohols" in *Chem. Anal. (Warsaw)*. 1993, 38:139-147.
118. Steinhanses, J. and K. Schoene. "Thermal Desorption-Gas Chromatography of some Organophosphates and S-Mustard after Trapping on Tenax" in *J. Chromatogr.* 1990, 514:273-278.
119. Syage, J. A. "Real-Time Detection of Chemical Agents using Molecular Beam Laser Mass Spectrometry" in *Anal. Chem.* 1990, 62:505A-509A.

120. Tingfa, D. "Gas Chromatographic Determination of O-Ethyl S-(N,N-Diisopropylamino)ethyl Methylphosphonothiolate and O,O-Diisopropyl S-Benzyl Phosphorothiolate as Corresponding Phosphonofluoridate and Phosphorofluoridate" in *Intern. J. Environ. Anal. Chem.* 1986, 27:151-158.
121. Tornes, A. F. "Identification of Some Alkyl Methylphosphonic Acids by Thermospray Mass Spectrometry" in *Rapid Commun. Mass Spectrom.* 1996, 10:878-882.
122. Tornes, J. A. and B. A. Johnsen. "Gas Chromatographic Determination of Methylphosphonic Acids by Methylation with Trimethylphenylammonium Hydroxide" in *J. Chromatogr.* 1989, 467:129-138.
123. Tripathi, D. N. "Mass Spectrometric Identification of Methylphosphonic Acid: The Hydrolysis Product of Isopropyl Methylphosphonofluoridate and Pinacolyl Methylphosphonofluoridate" in *Anal. Chem.* 1992, 64:823-824.
124. Tripathi, D. N., A. Bhattacharya and R. Vaidyanathaswamy. "Mass Spectra Identification of Bis(2-chloroethyl)sulfide and Related Compounds" in *Can. Soc. Forens. Sci. J.* 1984, 17:55-57.
125. Tripathi, D. N., M. P. Kaushik and A. Bhattacharya. "Gas Chromatographic-Mass Spectrometric Identification of a Mixture of Isopropyl Methylphosphonofluoridate, Pinacolyl Methylphosphonofluoridate and Diisopropyl Fluoro Phosphate" in *Can. Soc. Forens. Sci. J.* 1987, 2:151-153.

126. Tripathi, D. N., R. C. Malhotra and A. Bhattacharya. "Gas Chromatographic-Mass Spectrometric Identification of w-Chloroacetophenone, o-Chlorobenzylidenemalononitrile and Dibenz[b,f]-1:4-oxazepine" in *J. Chromatogr.* 1984, 315:417-419.
127. Verweij, A. and H. L. Boter. "Degradation of S-2-Diisopropylaminoethyl O-Ethyl Methylphosphonothioate in Soil: Phosphorus containing Products" in *Pestic. Sci.* 1976, 7:355-362.
128. Verweij, A., E. Burghardt and A. W. Koonings. "Gas Chromatographic Separation of Diastereoisomeric Alkyl Methylphosphonofluoridates and Related Compounds" in *J. Chromatogr.* 1971, 54:151-156.
129. Verweij, A., W. H. Dekker, H. C. Beck and H. L. Boter. "Hydrolysis of some Methylphosphonites and Methylphosphinates" in *Analytica Chimica Acta.* 1983, 151:221-225.
130. Vincze, A., K. L. Busch and R. G. Cooks. "Secondary Ion Mass Spectra of Quaternary Pyridine Aldoximes" in *Analytica Chimica Acta.* 1982, 136:143-153.
131. Vycudilik, W. "Detection of Bis(2-chloroethyl)-Sulfide (Yperite) in Urine by High Resolution Gas Chromatography-Mass Spectrometry" in *Forensic Sci. Intern.* 1987, 35:67-71.
132. Ward, J. R., J. W. Hovanec, J. M. Albizo, L. L. Szafraniec and W. T. Beaudry. "Decomposition of Phosphonofluoridates on Glass" in *J. Fluorine Chem.* 1991, 51:277-282.

133. Wensing, M. W., A. P. Snyder and C. S. Harden. "Energy Resolved Mass Spectrometry of Diethyl Alkyl Phosphonates with an Atmospheric Pressure Ionization Tandem Mass Spectrometer" in *J. Mass Spectrom.* 1995, 30:1539-1545.
134. Wensing, M. W., A. P. Snyder and C. S. Harden. "Energy Resolved Mass Spectrometry of Dialkyl Methylphosphonates with an Atmospheric Pressure Ionization Tandem Mass Spectrometer" in *Rapid Commun. Mass Spectrom.* 1996, 10:1259-1265.
135. Wils, E. R. J. "Mass Spectral Data of Precursors of Chemical Warfare Agents" in *Fresenius J. Anal. Chem.* 1990, 338:22-27.
136. Wils, E. R. J., A. G. Hulst, A. L. de Jong, A. Verweij and H. L. Boter. "Analysis of Thiodiglycol in Urine of Victims of an Alleged Attack with Mustard Gas" in *J. Anal. Toxicology.* 1985, 9:254-257.
137. Wils, E. R. J., A. G. Hulst and J. van Laar. "Analysis of Thiodiglycol in Urine of Victims of an Alleged Attack with Mustard Gas, Part II" in *J. Anal. Toxicology.* 1988, 12:15-19.
138. Wils, E. R. J. and A. G. Hulst. "Determination of Organophosphorus Acids by Thermospray Liquid Chromatography-Mass Spectrometry" in *J. Chromatogr.* 1988, 454:261-272.
139. Wils, E. R. J. and A. G. Hulst. "Determination of O-Ethyl S-2-Diisopropylaminoethyl Methylphosphonothioate (VX) by Thermospray Liquid Chromatography-Mass Spectrometry" in *J. Chromatogr.* 1990, 523:151-161.



140. Wils, E. R. J., A. G. Hulst and A. L. de Jong. "Determination of Mustard Gas and Related Vesicants in Rubber and Paint by Gas Chromatography-Mass Spectrometry" in *J. Chromatogr.* 1992, 625:382-386.
141. Wils, E. R. J. and A. G. Hulst. "Gas Chromatographic-Mass Spectrometric Identification of Tear-Gases in Dilute Solutions using Large Injection Volumes" in *J. Chromatogr.* 1985, 330:379-382.
142. Wils, E. R. J., A. G. Hulst, P. E. J. Verwiël, S. H. van Krimpen and A. Niederhauser. "Identification of an Octyl Methylphosphonofluoridate Mixture in Chemical Industry Samples" in *Fresenius J. Anal. Chem.* 1992, 343:297-303.
143. Wils, E. R. J. and A. G. Hulst. "Mass Spectra of some Derivatives of the Irritant o-Chlorobenzylidenemalononitrile" in *Fresenius Z. Anal. Chem.* 1985, 320:357-360.
144. Wils, E. R. J. and A. G. Hulst. "Mass Spectra of some Derivatives of 2,2'-Dichlorodiethyl Sulphide (Mustard Gas)" in *Fresenius Z. Anal. Chem.* 1985, 321:471-474.
145. Wils, E. R. J. and A. G. Hulst. "Mass Spectra of some Pinacolyl-Containing Organophosphorus Compounds" in *Org. Mass Spectrom.* 1986, 21:763-765.
146. Wils, E. R. J. and A. G. Hulst. "Thermospray Mass Spectrometry of Diquaternary Pyridinium Oxime Salts" in *Biomed. Environ. Mass Spectrom.* 1988, 17:155-159.
147. Wils, E. R. J. and A. G. Hulst. "The use of Thermospray-Liquid Chromatography/Mass Spectrometry for the Verification of Chemical Warfare Agents" in *Fresenius J. Anal. Chem.* 1992, 342:749-758.

148. Witkiewicz, Z., M. Mazurek and J. Szulc. "Chromatographic Analysis of Chemical Warfare Agents" in *J. Chromatogr.* 1990, 503:293-357.
149. Woloszyn, T. F. and P. C. Jurs. "Quantitative Structure-Retention Relationship Studies Of Sulfur Vesicants" in *Anal. Chem.* 1992, 64:3059-3063.
150. Yang, Y-C, J. A. Baker and J. R. Ward. "Decontamination of Chemical Warfare Agents" in *Chem. Rev.* 1992, 92:1729-1743.

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